

United States Patent 5,905,063  
Tanabe, et al. May 18, 1999

Tanabe teaches "A remover solution composition for resist which comprises (a) a salt of hydrofluoric acid with a metal-free base, (b) a water-soluble organic solvent, and (c) water and optionally (d) an anticorrosive, and has a pH or 5 to 8."

Tanabe teaches in claim 3, "The remover solution composition for resist as claimed in claim 1, wherein component (a) is ammonium fluoride"

Tanabe teaches in claim 2, "The remover solution composition for resist as claimed in claim 1, wherein component (a) is a salt of hydrofluoric acid with at least one metal-free base selected from the group consisting of hydroxylamines; primary, secondary, or tertiary aliphatic alicyclic, aromatic and heterocyclic amines; ammonia water; and C.sub.1 to C.sub.4 lower alkyl quaternary ammonium salt groups"

Tanabe teaches the water-soluble solvent in claim 5, "a water-soluble organic solvent which is at least one solvent selected from the group consisting of dimethyl sulfoxide, 3,3-dimethyl-2-imidazolidinone, and diethylene glycol monobutyl ether"

Tanabe generally teaches the water-soluble solvent to be "Examples of the water-soluble organic solvents include sulfoxides, sulfones, amides such as N,N-dimethylacetamide, lactams such as N-methyl-2-pyrrolidone, imidazolidinones, lactones, and polyhydric alcohols (e.g., ethylene glycol, ethylene glycol inonomethyl ether, ethylene glycol monoethyl ether, ethylene glycol monobutyl ether, ethylene glycol, etc."

Tanabe teaches corrosion inhibitors in amounts ranging from 0.5 to 15% including triazole compounds such as benzotriazole, o-tolyltriazole, m-tolyltriazole, p-tolyltriazole, carboxybenzotriazole, 1-hydroxybenzotriazole, nitrobenzotriazole, and dihydroxypropylbenzotriazole. Among these, preferred is benzotriazole.

Tanabe teaches weight percentages of the individual components in the following ranges: "In the case where the composition of the present invention comprises components (a) to (d), it is desirable that the content of component (a) is from 0.2 to 8% by weight, preferably from 0.5 to 5% by weight; the content of component (b) is from 40 to 80% by weight, preferably from 55 to 75% by weight; the content of component (d) is from 0.5 to 15% by weight, preferably from 0.5 to 10% by weight; and the remainder is component (c).

If the amount of each component is outside the range shown above, the composition is impaired in the removability of modified films and in noncorrosive properties. "

-----claim tree-----

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-----best-----

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4592787  
5968848  
5905063  
5792274  
4395348  
4765844  
4617251  
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510/176  
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430/331  
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510/202  
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430/329  
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216/48  
252/793  
430/309  
430/256  
252/396  
510/164  
510/413  
510/188  
510/204  
438/906  
510/493  
430/258  
438/745  
510/407  
510/422  
508/208  
510/500  
134/41  
510/504

-----keywords-----

fluoroboric acid ammonium fluoride alkaline ph ammonium fluoride tetramethyl ammonium fluoride tetraethyl amine sulfoxide sulfone amide lactone pyrrolidone imidazolidinone glycol glycol ether propylene glycol ethylene glycol nmp methylpyrrolidone pyrrolidone pyrrolidione ammonium hydroxide benzotriazole vanillin hepes weak acid weak acid weak benzotriazole ammonium hydroxide hydroxide ethylene glycol propylene glycol propylene glycol ether pyrrolidone lactone amide tetraethyl tetramethyl ammonium tetramethyl alkaline ammonium fluoride fluoride fluoroboric acid buffered fluoroboric sulfoxide sulfone imidazolidinone nmp aqueous acid ammonium amine glycol ether ethylene methylpyrrolidone pyrrolidione vanillin hepes

-----references-----

----- 5185235  
classes:1 430/331 1 134/40 1 134/42 1 216/48 1 430/309 1 430/325 1 430/329 1 510/176 1 510/202 1 510/204 1 510/212 1 510/504  
score: 826

keywords: glycol;propylene glycol;ethylene glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;ethylene glycol;propylene glycol;propylene;tetramethyl ammonium;tetramethyl;aqueous;ammonium;glycol;ether;ethylene;

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United States Patent: 5,185,235  
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( 1of1)

United States Patent  
5,185,235

Sato

, &nbsp;et al.

February 9, 1993

Remover solution for photoresist

Abstract

The remover solution of the invention comprises (A) from 35 to 80% by weight of an alcoholic solvent such as \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, (B) from 10 to 40% by weight of an organic solvent which is a halogenated hydrocarbon solvent, e.g., 1,2-dichlorobenzene and methylene chloride, an \*\*ether\*\* solvent, e.g., tetrahydrofuran, or an aromatic solvent, e.g., benzene and xylene, and (C) from 0.1 to 25% by weight of a quaternary \*\*ammonium\*\* compound such as \*\*tetramethyl ammonium \*\*hydroxide\*\* and trimethyl hydroxyethyl \*\*ammonium \*\*hydroxide\*\*. Different from conventional remover solutions which only can swell cured photoresist compositions, the inventive remover solution has a power to completely dissolve a cured photoresist layer

- sist which consists essentially of, in admixture:

(A) an aliphatic alcoholic solvent selected from the group consisting of methyl alcohol, ethyl alcohol, propyl alcohol, 3-methyl-3-methoxy butyl alcohol, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\* monomethyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\* in a weight fraction in the range from 35% to 80%;

(B) an organic solvent selected from the group consisting of halogenated hydrocarbon solvents, unhydroxylated \*\*ether\*\* solvents selected from the group consisting of dioxane, tetrahydrofuran, diethylene \*\*glycol\*\* dimethyl \*\*ether\*\*, triethylene \*\*glycol\*\* dimethyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* dimethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* diethyl \*\*ether\*\*, diethylene \*\*glycol\*\* diethyl \*\*ether\*\* and diethylene \*\*glycol\*\* dibutyl \*\*ether\*\*, and unhalogenated aromatic solvents in a

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classes:1 134/38 1 510/176 1 510/413 1 510/421 1 510/422 1 510/435  
score: 704

keywords: alkaline;ph;glycol;glycol ether;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol;propylene;glycol ether;alkaline;aqueous;acid;ammonium;glycol;ether;ethylene;

- tants may be used. It is further taught that it may be desirable to add up to 10 percent of an \*\*alkaline\*\* agent.

In the many heretofore known stripping compositions for stripping photoresist, there are components which are toxic, corrosive, or are hazardous to use. Furthermore, many of these stripping compositions do not strip the photoresist in an expedient manner.

What is needed is a composition useful in stripping photoresist polymers from substrates which does not contain toxic reagents, does not contain corrosive agents, and does not contain reagents which are hazardous to use. Furthermore, what is needed is a composition which results in faster stripping of the photoresist polymer mask.

#### SUMMARY OF THE INVENTION

This invention is a composition suitable for stripping cross-linked photoresist polymer which comprises

- (a) a lower alkyl monoether of a \*\*propylene glycol\*\*, or a mixture of lower alkyl monoethers of \*\*propylene glycol\*\*;

- (b) a

- t the photoresist polymer from the substrate, so as to break up the coating. Further, the lower alkyl monoethers of \*\*propylene glycol\*\* serve as cosolvents for the photoresist polymer. Preferred lower alkyl monoethers of \*\*propylene glycol\*\* correspond to the formula

##STR1##  
wherein

R.sup.1 is C.sub.1-4 alkyl;

R.sup.2 is hydrogen or methyl; and

n is an integer of 1 to 3,

with the proviso that one R.sup.2 in each unit is methyl and the other is hydrogen. Unit refers herein to the moiety

##STR2##

Examples of lower alkyl monoethers of \*\*propylene glycol\*\* useful in this invention include \*\*propylene glycol\*\* methylether, \*\*propylene glycol\*\* ethylether, \*\*propylene glycol\*\* propylether, \*\*propylene glycol\*\* isopropylether, \*\*propylene glycol\*\* butylether, \*\*propylene glycol\*\* isobutylether, dipropylene glycol methylether, dipropylene glycol ethylether, dipropylene glycol propylether, dipropylene glycol isopropylether, dipropylene glycol butylether, dipr

- opylene glycol isobutylether, tripropylene glycol methylether, tripropylene glycol ethylether, tripropylene glycol propylether, tripropylene glycol isopropylether, tripropylene glycol butylether and tripropylene glycol isobutylether. More preferred lower alkyl monoethers of \*\*propylene glycol\*\* include \*\*propylene glycol\*\* methylether, \*\*propylene glycol\*\* ethylether, \*\*propylene glycol\*\* isopropylether, \*\*propylene glycol\*\* propylether, dipropylene glycol methylether, dipropylene glycol propylether, dipropylene glycol isopropylether and tripropylene glycol methylether. Most preferred are \*\*propylene glycol\*\* isopropylether and \*\*propylene glycol\*\* methylether.

Any C.sub.2-6 alkanol is useful in this invention. The C.sub.2-6 alkanol functions as a penetrating solvent and is a good wetting agent for the substrate. Examples of C.sub.2-6 alkanols useful in this invention are ethanol, propanol, isopropanol, n-butanol, isobutanol, pentane and hexane.

Pre

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classes:1 438/745 1 134/2 1 134/3 1 134/41 1 438/906 1 510/176  
score: 699

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;glycol;propylene glycol;ethylene glycol;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene glycol;propylene;alkaline;ammonium fluoride;fluoride;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- g a hydrofluorate remover solution, and is a composition containing \*\*ethylene glycol\*\* and/or \*\*propylene glycol\*\* and another water-soluble organic solvent.

The water-soluble organic solvent is a water-soluble organic solvent other than \*\*ethylene glycol\*\* and \*\*propylene glycol\*\*, and examples of such solvents include:

monohydric alcohols such as methyl alcohol, ethyl alcohol, and isopropyl alcohol;

ketones such as acetone;  
 sulfoxides such as dimethyl \*\*sulfoxide\*\*;  
 sulfones such as dimethyl \*\*sulfone\*\*, diethyl \*\*sulfone\*\*, bis(2-hydroxyethyl)  
 \*\*sulfone\*\*, and \*\*tetramethyl\*\*ene \*\*sulfone\*\*;  
 amides such as N,N-dimethylformamide, N-methylformamide,  
 N,N-dimethylacetamide, N-methylacetamide, and N,N-diethylacetamide;  
 lactams such as N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone,  
 N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, and  
 N-hydroxyethyl-2-pyrrolidone;  
 imidazolidinones such as 1,3-dimethyl-2-imidazolidinone,  
 1,3-diethyl-2-imidazolidinone, and 1,3-diisopropyl-2-  
 butyric acid,  
 isobutyric acid, oxalic acid, malonic acid, succinic acid, glutaric acid,  
 maleic acid, fumaric acid, benzoic acid, phthalic acid,  
 1,2,3-benzenetricarboxylic acid, \*\*glycol\*\*ic acid, lactic acid, malic acid,  
 citric acid, acetic anhydride, phthalic anhydride, maleic anhydride,  
 succinic anhydride, and salicylic acid. Formic acid, phthalic acid,  
 benzoic acid, phthalic anhydride, and salicylic acid are the preferred  
 carboxyl-group-containing organic compounds. Phthalic anhydride and  
 salicylic acid are especially preferred.  
 Examples of triazole compounds include \*\*benzotriazole\*\*, o-tolyltriazole,  
 m-tolyltriazole, p-tolyltriazole, carboxybenzotriazole, 1-hydroxytriazole,  
 nitrobenzotriazole, and dihydroxypropylbenzotriazole. Among these,  
 \*\*benzotriazole\*\* is preferred.  
 Typical examples of saccharides include D-sorbitol, arabitol, mannitol,  
 sucrose, and starch. Among these, D-sorbitol is preferred.  
 Each of the above-listed anticorr  
 - could not be observed,  
 \*\*ammonium \*\*fluoride\*\* salt was deposited during the rinse treatment.

TABLE 1

Composition of Rinse solution (weight ratio)	Degree of Corrosion after Dipping in Water at 23.degree. C. for 20 min.
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## Example 1

Methanol:Ethylene **glycol**	Good
(50:50)	
Example 2 Methanol:Ethylene **glycol**	Good
(80:20)	
Example 3 Ethanol:Ethylene **glycol**	Good
(70:30)	
Example 4 Ethanol:Ethylene **glycol**:	Good
Pyrocatechol (30:68:2)	
Example 5 Isopropyl Alcohol:Ethylene	Good
**glycol** (70:30)	
Example 6 Isopropyl Alcohol:Ethylene	Good
**glycol** (50:50)	
Example 7 Methanol:Propylene	

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 classes:1 510/176 1 134/13 1 134/2 1 134/3 1 134/40 1 134/42 1 510/255 1 510/258 1 510/264 1 510/269 1 51  
 0/401 1 510/402  
 score: 682

keywords: ammonium fluoride;ph;ammonium fluoride;sulfoxide;glycol;ethylene glycol;nmp;hydroxide;ethylene  
 glycol;ammonium fluoride;fluoride;buffered;sulfoxide;nmp;aqueous;acid;ammonium;amine;glycol;ether;ethylen  
 e;

- lpiperidine, oxazole, and thiazole. Examples of the C.sub.1  
 to C.sub.4 lower alkyl quaternary \*\*ammonium\*\* salt groups include  
 \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* and trimethyl(2-hydroxyethyl)-ammonium  
 \*\*hydroxide\*\* (choline). Preferred metal-free bases are ammonia water,  
 monoethanolamine, and \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\*, because these bases  
 are easily available and safe. A salt of the metal-free base with  
 hydrofluoric acid can be produced by adding the metal-free base to  
 commercial hydrofluoric acid having a hydrogen \*\*fluoride\*\* concentration of  
 50 to 60% in such a proportion as to result in a pH of 5 to 8. Commercial  
 \*\*ammonium \*\*fluoride\*\* is, of course, usable as the salt.

The composition of the present invention has an almost neutral pH value of  
 5 to 8. This pH range may be attained by using component (a) which is

almost neutral. The proportion of hydrofluoric acid to a metal-free base to be added thereto so as to attain a neutral pH value varies with the treatments, which are necessary in resist removing processes where hydrogen fluoride generates.

- Any organic solvent may be used as component (b) in the present invention, as long as it is miscible with components (a), (c), and (d). Water-soluble organic solvents used in conventional organic amine-based remover solution compositions may be used. Examples of the water-soluble organic solvents include sulfoxide (e.g., dimethyl sulfoxide), sulfone (e.g., dimethyl sulfone, diethyl sulfone, bis(2-hydroxyethyl) sulfone, tetramethylethylene sulfone), amide (e.g., N,N-dimethylformamide, N-methylformamide, N,N-dimethylacetamide, N-methylacetamide, N,N-diethylacetamide), lactams (e.g., N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, N-hydroxyethyl-2-pyrrolidone), imidazolidinone (e.g., 1,3-dimethyl-2-imidazolidinone, 1,3-diethyl-2-imidazolidinone, 1,3-diisopropyl-2-imidazolidinone), lactone (e.g., gamma-butyrolactone, delta-valerolactone), and polyhydric alcohols (e.g., ethylene glycol, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol monobutyl ether, ethylene glycol monomethyl ether acetate, diethylene glycol, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, diethylene glycol monobutyl ether); and derivatives thereof. Among these, preferred are dimethyl sulfoxide, N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, ethylene glycol, and diethylene glycol monobutyl ether, because these solvents bring about the removability of modified resist films. In particular, use of a water-soluble organic solvent comprising at least 10% by weight (wt %) ethylene glycol as component (b) is preferred in that the composition of the present invention contains

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classes: 1 134/13 1 134/2 1 134/3 1 134/40 1 134/42 1 510/176 1 510/255 1 510/258 1 510/264 1 510/269 1 510/401 1 510/402  
score: 652

keywords: ammonium fluoride;ph;ammonium fluoride;sulfoxide;glycol;ethylene glycol;nmp;hydroxide;ethylene glycol;ammonium fluoride;fluoride;buffered;sulfoxide;nmp;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- and dicyclohexylamine. Examples of the aromatic amine include benzylamine, dibenzylamine, and N-methylbenzylamine. Examples of the heterocyclic amine include pyrrole, pyrrolidine, pyrrolidone, pyridine, morpholine, pyrazine, piperidine, N-hydroxyethylpiperidine, oxazole, and thiazole. Examples of the C.sub.1 to C.sub.4 lower alkyl quaternary ammonium salt groups include tetramethylammonium hydroxide and trimethyl(2-hydroxyethyl)-ammonium hydroxide (choline). Preferred metal-free bases are ammonia water, monoethanolamine, and tetramethylammonium hydroxide, because these bases are easily available and safe. A salt of the metal-free base with hydrofluoric acid can be produced by adding the metal-free base to commercial hydrofluoric acid having a hydrogen fluoride concentration of 50 to 60% in such a proportion as to result in a pH of 5 to 8. Commercial ammonium fluoride is, of course, usable as the salt.

The composition of

- resist and inhibiting the composition from corroding a metal film on a substrate or peripheral devices including a remover feeder. Furthermore, a low hydrogen fluoride content of component (a) eliminates the necessity of troublesome exhaust and wastewater treatments, which are necessary in resist removing processes where hydrogen fluoride generates.
- Any organic solvent may be used as component (b) in the present invention, as long as it is miscible with components (a), (c), and (d). Water-soluble organic solvents used in conventional organic amine-based remover solution compositions may be used. Examples of the water-soluble organic solvents include sulfoxide (e.g., dimethyl sulfoxide), sulfone (e.g., dimethyl sulfone, diethyl sulfone, bis(2-hydroxyethyl) sulfone, tetramethylethylene sulfone), amide (e.g., N,N-dimethylformamide, N-methylformamide, N,N-dimethylacetamide, N-methylacetamide, N,N-diethylacetamide), lactams (e.g., N

- -methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, N-hydroxyethyl-2-pyrrolidone), \*\*imidazolidinone\*\*s (e.g., 1,3-dimethyl-2-imidazolidinone, 1,3-diethyl-2-imidazolidinone, 1,3-diisopropyl-2-imidazolidinone), \*\*lactone\*\*s (e.g., .gamma.-butyrolactone, .delta.-valerolactone), and polyhydric alcohols (e.g., \*\*ethylene glycol\*\*, \*\*ethylene glycol\*\* monomethyl \*\*ether\*\*, \*\*ethylene glycol\*\* monoethyl \*\*ether\*\*, \*\*ethylene glycol\*\* monobutyl \*\*ether\*\*, \*\*ethylene glycol\*\* monomethyl \*\*ether\*\* acetate, \*\*ethylene glycol\*\* monoethyl \*\*ether\*\* acetate, diethylene glycol, diethylene glycol monomethyl \*\*ether\*\*, diethylene glycol monoethyl \*\*ether\*\*, diethylene glycol monobutyl \*\*ether\*\*); and derivatives thereof. Among these, preferred are dimethyl \*\*sulfoxide\*\*, N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, \*\*ethylene glycol\*\*, and diethylene glycol monobutyl \*\*ether\*\*, because these solvents bring a

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classes:1 510/176 1 134/3 1 134/38 1 134/40 1 134/41 1 134/42 1 430/329 1 430/331 1 510/202 1 510/414 1 510/505 1 568/763  
score: 635

keywords: glycol;ethylene glycol;ethylene glycol;fluoride;acid;glycol;ethylene;

- ortho, meta and para isomers of xylene and diethylbenzene; trialkyl-substituted aromatic hydrocarbons containing 9 to 20 carbon atoms, such as the 1,2,3-; 1,2,4- and 1,3,5-isomers of trimethyl and triethylbenzene; suitable polar organic solvents include aliphatic ketones containing three to 10 carbon atoms, such as acetone, methylethylketone and methylisobutylketone; monoalkyl \*\*ether\*\*s of \*\*ethylene glycol\*\* containing three to 10 carbon atoms, such as ethoxyethanol and butoxyethanol; carboxylic acids containing one to four carbon atoms, such as acetic and maleic acid; formamide; N,N-dialkylalkanonylamides containing three to 10 carbon atoms, such as dimethylformamide and dimethylacetamide; N-alkyl lactams containing six to 12 carbon atoms, such as N-methylpyrrolidone; cyclic aliphatic \*\*sulfone\*\*s containing four to 6 carbon atoms, such as \*\*tetramethyl\*\*enesulfone; and the like. The preferred nonpolar, organic solvents are xylene

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classes:1 510/176 1 134/38 1 252/364 1 430/329 1 510/202 1 510/212 1 510/499 1 510/506  
score: 628

keywords: glycol;glycol ether;propylene glycol;ethylene glycol;ammonium hydroxide;ammonium hydroxide;hydrate;ethylene glycol;propylene glycol;propylene;glycol ether;tetramethyl ammonium;tetramethyl;aqueous;ammonium;amine;glycol;ether;ethylene;

United States Patent: 4,765,844

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( 10fl)

United States Patent

4,765,844

Merrem

, &nbsp; et al.

August 23, 1988

Solvents for photoresist removal

Abstract

The invention relates to solvent systems which are based on water-soluble amino derivatives and \*\*propylene glycol\*\* components, for removing photoresists. These solvent systems are comprised of

- (a) from about 10 to 100% by weight of at least one water-soluble \*\*amine\*\* of the general formula

##STR1##

in which R.sup.1, R.sup.2, R.sup.3, R.sup.4 denote H or alkyl groups

n,m denote 0 to 2, and

o denotes 1 to 3; and

- (b) from about 0 to 90% by weight of at least one water-soluble \*\*propylene\*\*

**\*\*glycol\*\*** derivative of the general formula  
**##STR2##**  
 in which R.sup.3, R.sup.4 denote H, alkyl or  
**##STR3##**  
 and P denotes 1 to 3.

## Inventors:

Merrem; Hans-Joachim(Seeheim-Jugenheim, DE);  
 Schmitt; Axel(Walluf, DE)

## Assignee:

- the formula  
**##STR7##**

in which R.sup.1, R.sup.2, R.sup.3, R.sup.4 denote H or alkyl groups  
 n, m denote 0 to 2, and  
 o denotes 1 to 3;  
 and

(b) from about 10 to 90% by weight of at least one water-soluble **\*\*propylene\*\***  
**\*\*glycol\*\*** derivative represented by the formula  
**##STR8##**  
 in which R.sup.3, R.sup.4 denote H, alkyl or  
**##STR9##**  
 groups, and P denotes 1 to 3; wherein said agent is biodegradable, of low  
 toxicity, free of **\*\*ammonium \*\*hydroxide\*\*** base, relatively non corrosive to  
 aluminum and wherein substantially all components of said agent boil at  
 temperatures of at least 160.degree. C.

2. An agent as claimed in claim 1, wherein said water-soluble **\*\*amine\*\*** is  
 selected from the group consisting of isopropanolamine and  
 N-(2-aminoethyl)-ethanolamine.

3. An agent as claimed in claim 1, wherein said water-soluble **\*\*propylene\*\***  
**\*\*glycol\*\*** derivative is selected from the group consisting of dipropylene  
**\*\*glycol\*\*** methyl **\*\*ether\*\*** and tripropylene **\*\*glycol\*\*** met

- systems, based on  
 water-soluble amino derivatives and **\*\*propylene \*\*glycol\*\*** compounds, that are  
 useful for removing photoresists.

Photoresists are used as masks in the production of, via etching or  
 implantation processes, integrated component parts, circuit boards, and  
 the like. After having served their purposes, the masks are generally  
 removed, frequently by means of agents which, depending on their mode of  
 action, are known as "strippers" or "removers".

Systems of this kind are described, for example, by W. S. de Forest in  
 "Photoresist: Materials and Processes" pages 203 et seq., McGraw-Hill Book  
 Co., New York, 1975. They contain, e.g., chlorinated hydrocarbons, such as  
 methylenechloride; polar solvents, such as dimethylformamide,  
 N-methyl-2-pyrrolidone and monoethanolamine; **\*\*glycol \*\*ether\*\***s, such as  
**\*\*ethylene \*\*glycol\*\*** monoethyl **\*\*ether\*\***, **\*\*ethylene \*\*glycol\*\*** monobutyl **\*\*ether\*\*** and the  
 acetates thereof; strong bases, such as **\*\*tetramethyl ammonium\*\***

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classes:1 430/256 1 134/38 1 430/258 1 430/331 1 510/176 1 510/212 1 510/407 1 510/493 1 510/499 1 510/50  
 0 1 510/501 1 510/505  
 score: 620

keywords: glycol;nmp;nmp;aqueous;acid;amine;glycol;ether;

- pletion of the stripping operation by the use of  
**\*\*aqueous\*\*** materials, such as deionized water.

Now it has been discovered, in accordance with the present invention, that  
 the foregoing problems can be overcome with a stripping composition that  
 includes a select **\*\*amine\*\*** compound and an organic polar solvent, the  
 composition being essentially free of phenol compounds and halogenated  
 hydrocarbon compounds. In the practice of the invention, an organic  
 polymeric material is removed from a substrate by contacting the organic  
 polymeric material with the stripping composition as described above.  
 Surprisingly, it has been found that the stripping composition is highly  
 effective in removing organic polymeric materials from substrates,  
 although as noted it is essentially free of phenol compounds and  
 halogenated hydrocarbon compounds, the use of which is taught in the art.  
 Furthermore, because the stripping composition is essentially free of s



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classes:1 510/202 1 134/2 1 134/3 1 134/38 1 134/40 1 134/41 1 510/206 1 510/417 1 510/488 1 510/499 1 510/500 1 510/506  
score: 606

keywords: pyrrolidone;glycol;glycol ether;propylene glycol;ethylene glycol;nmp;pyrrolidone;ammonium hydroxide;ammonium hydroxide;hydroxide;ethylene glycol;propylene glycol;propylene;glycol ether;pyrrolidone;nmp;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- the proportions indicated. In the formulations of the present invention, the \*\*pyrrolidone\*\* constituent is believed to achieve an enhanced varnish removing or cleaning ability when combined with a \*\*propylene \*\*glycol\*\* aliphatic \*\*ether\*\* compound, including an acetate thereof. Such combinations of N-methyl-2-pyrrolidone and a \*\*propylene \*\*glycol\*\* solvent are utilized in the various formulations of the present invention.

The alkanolamine component of the present invention is preferably an ethanolamine and, most preferably, monoethanolamine. Monoethanolamine is an extremely effective solvent for varnish and the type of baked on carbon residue which is typical of various parts of internal combustion engines following substantial and continuous use under normal as well as extreme operating conditions. In the appropriate proportions, monoethanolamine will form an effective soap with one or more aliphatic acid in a single phase cleaning system in

- y be used with various aluminum alloys without damage thereto. Furthermore, the corrosive characteristics of such caustic mixtures has always been a danger to those using such formulations. By use of the present invention, the dangers of corrosive damage as well as other inherent health hazards are avoided.

Generally, the cleaning compositions of the present invention contain no chlorinated solvents. The alkanolamine is used to remove varnish and carbon deposits from various engine parts and will combine with aliphatic fatty acids, when present, to form a soap. \*\*ammonium \*\*hydroxide\*\* may be used in combination with the alkanolamine to further enhance carbon removal. The \*\*propylene glycol \*\*ether\*\*, or an ester thereof, combined with an alkylpyrrolidone, is included as a degreasing substitute for methylene chloride. In the formulations disclosed and described herein, the present invention is generally less volatile and less of a health

----- 6323169

classes:1 510/176 1 510/175 1 510/245 1 510/255 1 510/257 1 510/264 1 510/499  
score: 602

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;sulfone;amide;glycol;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol;propylene;amide;tetramethyl;alkaline;ammonium fluoride;fluoride;sulfone;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- anediaminetetramethylenephosphonic acid, diethylenetriaminepentamethylenephosphonic acid and \*\*ethylene\*\*diaminetetrakis(methylenephosphonic acid are preferable, and particularly preferred is 1,2-propanediaminetetramethylenephosphonic acid.

The above chelating agents may be used alone or in combination of two or more.

The concentration of the chelating agent preferably 0.01 to 5% by weight, more preferably 0.05 to 3% by weight based on the total weight of the stripping composition.

The water-soluble fluorine compounds (c) usable in the present invention may include organic \*\*amine fluoride\*\*s such as \*\*ammonium \*\*fluoride\*\*, acid \*\*ammonium \*\*fluoride\*\* and monoethanolamine \*\*fluoride\*\*, and \*\*tetramethyl ammonium\*\* \*\*fluoride\*\*. The concentration of the water-soluble fluorine compound is preferably 0.001 to 10% by weight, more preferably 0.005 to 5% by weight based on the total weight of the stripping composition.

The organic solvents (d) usable in the present invention may include \*\*ether\*\*

- solvents such as \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\* monomethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monoethyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*, diethylene \*\*glycol\*\* dimethyl \*\*ether\*\* and dipropylene \*\*glycol\*\* dimethyl \*\*ether\*\*;
- \*\*amide\*\* solvents such as formamide, monomethylformamide, dimethylformamide, monoethylformamide,

report09881552.txt

diethylformamide, acetamide, monoethylacetamide, dimethylacetamide, monoethylacetamide, diethylacetamide, N-methylpyrrolidone and N-ethylpyrrolidone; and sulfur compound solvents such as dimethyl \*\*sulfoxide\*\*, dimethyl \*\*sulfone\*\*, diethyl \*\*sulfone\*\*, bis(2-hydroxy) \*\*sulfone\*\* and \*\*tetramethyl\*\*ene \*\*sulfone\*\*. Preferred are dimethyl \*\*sulfoxide\*\*, N,N-dimethyl

----- 5563119  
classes:1 510/176 1 134/2 1 134/38 1 134/40 1 430/329 1 510/201 1 510/206 1 510/212 1 510/401 1 510/402  
score: 553

keywords: alkaline;ammonium hydroxide;ammonium hydroxide;hydroxide;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;aqueous;acid;ammonium;amine;

- or  
different than R.sub.3. Preferred inhibitors include catechol, pyrogallol, anthranilic acid, gallic acid, gallic esters, and the like.  
The alkanolamines which are useful in the compositions of the invention are the lower alkanol \*\*amine\*\*s which include monoethanolamine, diethanolamine, mono-, di- and tri-isopropanolamine, and the like. Preferred is monoethanolamine alone or in combination with other alkanolamines.  
The tetraalkylammonium \*\*hydroxide\*\* compounds which are useful in the invention are \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), \*\*tetraethyl ammonium\*\* \*\*hydroxide\*\*, diethyl dimethyl \*\*ammonium\*\* \*\*hydroxide\*\*, and the like. Advantageously, the hydrates of the \*\*hydroxide\*\* compounds are utilized. The most preferred \*\*hydroxide\*\* compound is TMAH.5H.sub.2 O.  
The preferred stripping composition consists essentially of about 73% by weight of monoethanolamine, about 18% by weight of water, about 5% by weight of inhibitor and about 4% by weight of TMAH.5H.s

----- 5962383  
classes:1 510/164 1 134/42  
score: 553

keywords: ph;pyrrolidone;glycol;glycol ether;propylene glycol;ethylene glycol;pyrrolidone;ammonium hydroxide;weak;ammonium hydroxide;hydroxide;ethylene glycol;propylene glycol;propylene;glycol ether;pyrrolidone;tetramethyl ammonium;tetramethyl;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- such  
as \*\*weak acid\*\*s and their conjugate bases, for example, acetic acid and \*\*ammonium\*\* acetate. Preferred for use as such components are acetic acid, boric acid, citric acid potassium biphthalate, mixtures of \*\*ammonium\*\* chloride and \*\*ammonium\*\* acetate, especially a 1:1 mixture of these two salts, and mixtures of acetic acid and ammonia and other \*\*amine\*\*s.  
The following examples are illustrative of the present invention and are not meant to, and should not be taken to, limit the scope of the invention.

#### EXAMPLE 1

An optical mold is selected that has been contaminated with a diethylene \*\*glycol\*\* bisallyl carbonate (DEGBAC) based monomer. The polymer is hardened on the external side of the mold and the mold is further contaminated with fingerprint oils and dirt. The contaminated mold is immersed in a solution of 2.5% \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\*, 15% potassium \*\*hydroxide\*\*, 15% sodium \*\*hydroxide\*\* and 67.5% water at 150 to 160.degree. F. (ca 65.d

----- 5202049  
classes:1 510/206 1 134/38 1 134/39 1 134/40 1 510/405 1 510/421 1 510/424 1 510/437 1 510/499 1 510/506  
score: 540

keywords: ph;glycol;ethylene glycol;ethylene glycol;acid;glycol;ether;ethylene;

- es Cited [Referenced By]  
U.S. Patent Documents  
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4678605Jul., 1987Geke et al.252/547.  
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Primary Examiner:Lieberman; Paul  
Assistant Examiner:Parks; William S.

Parent Case Text

This is a continuation of copending application Ser. No. 07/610,196 filed  
on Nov. 6, 1990 now abandoned.

Claims

What is claimed is:

1. A sealer finish remover composition comprising effective amounts of (a)  
a solvent selected from diethylamine, diethanolamine, monoethanolamine,  
\*\*ethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* phenyl \*\*ether\*\*, and  
mixtures thereof, (b) water, (c) about 4-8 weight % of a metasilicate, and  
(d) about 1-30 weight % of a linear, 6-10 carbon, organic compound  
selected from caprylic acid, n-o

----- 4921626  
classes:1 252/794 1 216/90 1 216/99 1 252/793  
score: 528

keywords: ph;glycol;propylene glycol;weak;propylene glycol;propylene;fluoride;buffered;acid;ammonium;glyc  
ol;ethylene;

- 63.  
4585514Apr., 1986Scallan156/663.  
Primary Examiner:Powell; William A.  
Attorney, Agent or Firm:Lamb; Charles G.

Claims

What is claimed:

1. An etching composition for etching glass comprising:  
from about 16% to about 30% by weight of \*\*ammonium\*\* bifluoride;  
from about 2% to about 4% by weight of xanthum gum;  
from about 45% to about 60% by weight of water; and,  
from about 20% to about 24% by weight of \*\*propylene \*\*glycol\*\*.
2. The etching composition of claim 1, further comprising less than 2% by  
weight of phosphoric acid.
3. The etching composition of claim 2, having a pH of about 5.0.
4. A process for making a glass etching composition comprising the steps  
of:  
adding \*\*ammonium\*\* bifluoride to water in a first vessel while agitating the  
water;  
continuing to agitate the \*\*ammonium\*\* bifluoride-water mixture until all of  
the \*\*ammonium\*\* bifluoride is completely dissolved in the water;  
adding xanthum gum to \*\*propylene \*\*glycol\*\* while agitating the \*\*propylene \*\*glycol\*\*  
in a se

----- 4250193  
classes:1 514/772 1 424/701 1 424/73 1 514/777  
score: 522

keywords: glycol;glycol ether;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol  
;propylene;glycol ether;aqueous;acid;amine;glycol;ether;ethylene;

- 1 compounds  
(A).about.(H) underwent no substantial hydrolysis. To the contrary, both  
(I) and (J) had at least more than 90% hydrolyzed. In this connection, it  
has been found that the hydrolysis of each compound other than the  
compounds (A).about.(H) is in a similar order

Some preparation examples of the novel compounds of the present invention  
will now be given.

Preparation Example 1

0.5 grams of a boron trifluoride \*\*ether\*\* complex were added to 31 grams of  
\*\*ethylene \*\*glycol\*\* and were fully mixed under agitation therewith. To the  
resultant mixture were gradually added dropwise 102 grams of ethylglycidyl  
\*\*ether\*\* at room temperature over a period of 9.5 hours. The resultant  
product was then heated at about 100.degree. C. for 3 hours and was added  
with 10 grams of anhydrous sodium carbonate, followed by sufficient  
stirring and mixing. Thereafter, the undissolved matters were filtered  
off. The formed oily liquid was rectified under reduc

- 114 grams of 2-propenylglycidyl **ether** at room temperature over a period of 10 hours. The resultant product was then heated at about 100.degree. C. for 3 hours and was added with 50 cc of potassium carbonate solution, followed by sufficient stirring and mixing. Thereafter, the formed product from which the **aqueous** phase had been removed was rectified under reduced pressures to obtain 68 grams of bis-(2-propenylglyceryl)-tetramethylene **ether**.

## Preparation Example 4

- 0.5 grams of sodium **hydroxide** were added to 51 grams of diethylene **glycol** and were fully mixed under agitation therewith. To the resultant mixture were gradually added dropwise 102 grams of ethylglycidyl **ether** at room temperature over a period of 9.5 hours. The resultant product was then heated at about 100.degree. C. for 3 hours and was added with 10 grams of anhydrous sodium carbobate, followed by sufficient stirring and mixing. Thereafter the undissolved matters were
- ith. To the resultant mixture were gradually added dropwise 130 grams of butylglycidyl **ether** at 100.degree. C. over a period of 9.5 hours. The resultant product was at room temperature and was added with 100.degree. cc of sodium carbonate solution, followed by sufficient stirring and mixing. Thereafter, the **aqueous** phase had been removed. The formed oily liquid was rectified under reduced pressures to obtain 102 grams of bis-(butylglyceryl)-triethylene **glycol ether**.

## Preparation Example 7

- 0.5 grams of sodium **hydroxide** were added to 82 grams of **tetraethyl** **ene glycol** and were sufficiently mixed under agitation therewith. To the resultant mixture were gradually added dropwise 128 grams of amylglycidyl **ether** at 100.degree. C. over a period of 10 hours. The resultant product was at room temperature and was added with 50 cc of water, followed by sufficient stirring and mixing. Thereafter, the formed product from which the **aqueous**

- Bis-(propylglyceryl)-triethylene **ether**

	5
Polyoxyethylene-cured castor oil derivative	
	0.4
Perfume	0.1
Component B	
Refined water	20
Succinic acid	0.25
Sodium succinate	0.05
Component C	
Refined water	54.2

To the components A dissolved at 60.degree.-70.degree. C. were gradually added the components B at room temperature. The refined water C was then added to the resultant solution, followed by filtration at room temperature. Thus the product was formed.

## EXAMPLE 2--COSMETIC REMOVER

Component A	
Ethanol	15
<b>propylene glycol</b>	5
Bis-(propylglyceryl)-tetraethylene <b>ether</b>	
	10
Polyoxyethylene-cured castor oil derivative	

----- 6191086

classes:1 510/175 1 134/12 1 134/13 1 134/3 1 134/2214 1 510/176 1 510/178 1 510/201 1 510/202 1 510/203 1 510/204 1 510/210 1 510/212 1 510/225 1 510/245 1 510/254 1 510/256 1 510/257 1 510/259 1 510/412 1 510/477 1 510/480 1 510/504  
score: 518

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;tetramethyl ammonium fluoride;glycol;ethylene glycol;nmp;ammonium hydroxide;weak;ammonium hydroxide;hydroxide;ethylene glycol;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;ammonium fluoride;fluoride;nmp;aqueous;acid;ammonium;glycol;ethylene;

- reof. Preferred acid **fluoride**s include hydrogen **fluoride**, (HF), and perfluoric acid. Preferred **fluoride** salts are **ammonium fluoride**, **ammonium bifluoride**, **ammonium difluoride** and tetraalkyl **ammonium fluoride** salts such as **tetramethyl ammonium fluoride** and **tetraethyl ammonium fluoride**. Preferably, the fluorine-containing compound will comprise about

0.5 to 10 wt. % based upon the weight of the composition.

The polyammonium salts may be prepared by reacting a polyamine with HF to form the polyammonium \*\*fluoride\*\* salt. For example, HF may be reacted with an \*\*aqueous\*\* solution of \*\*ethylene\*\* diamine to form an \*\*ethylene\*\*diammonium difluoride. Alternatively, an excess amount of HF may be reacted with the polyamine to form the polyammonium salt. Preferred polyammonium salts include \*\*ethylene\*\*diammonium difluoride and diethylenetriammonium trifluoride.

It is believed that the fluorine-containing compound aids in removal of contaminants by slight

- amine salt has been found by the present applicants to be particularly useful as a cleaning composition.

The pH of the cleaning composition preferably ranges from weakly acidic to slightly basic so as to not attack or corrode the metal used in the microcircuit fabrication. If the cleaning composition is either too caustic or too acidic, the metal layers on the substrate are subject to attack. Therefore, the components in the cleaning composition should be admixed in appropriate concentrations to provide a composition having a pH with a preferred range from about 2 to 9, and more preferably from about 2 to 6.

In a preferred embodiment of this invention, the fluorine-containing compound is admixed with a composition comprising water, a hydroxylamine salt and at least one basic compound selected from the group consisting of: \*\*amine\*\*s, quaternary \*\*ammonium \*\*hydroxide\*\*s and \*\*ammonium \*\*hydroxide\*\*. Such compositions are described in co

----- 4822723  
classes:1 430/331 1 430/302 1 430/309 1 510/171 1 510/176 1 510/206  
score: 513

keywords: glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;aqueous;acid  
;amine;glycol;ether;ethylene;

- manufacture of lithographic printing plates such as, Alcoa 3003 and Alcoa 1100, which may or may not have been pretreated by standard graining and/or etching and/or anodizing techniques as are well known in the art, and also may or may not have been treated with a composition, such as polyvinyl phosphonic acid, suitable for use as a hydrophilizing layer for lithographic plates is coated with a light sensitive polymeric diazonium salt or photopolymer containing composition. Such compositions may also contain binding resins, such as polyvinyl formal resins, colorants, acid stabilizers, surfactants, exposure indicators or other art recognized ingredients. The photosensitive sheet material is then exposed to a suitable radiation source through a mask or transparency, and the exposed sheet then developed for removal of the non-image photosensitive materials. The photosensitive coating mixture is usually prepared in a solvent

- hem, to remove the non-image areas, using a developer which contains the aforementioned ingredients in \*\*aqueous\*\* mixture.

The developer contains a \*\*propylene \*\*glycol\*\* monomethyl \*\*ether\*\* or dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\* component. In the preferred embodiment this component is present in an amount of from about 25.0% to about 75.0% by weight of the developer. A more preferred range is from about 35.0% to about 65.0% and most preferably from about 45.0% to about 55.0%.

The developer also contains from about 0.05% to about 5.0% by weight of the developer of \*\*ethylene\*\* carbonate or \*\*propylene\*\* carbonate. A more preferred range is from about 0.1% to about 1.0% and most preferably from about 0.3% to about 0.7%.

The developer further contains from about 1.0% to about 30.0% by weight of the developer of \*\*ethylene \*\*glycol\*\* diacetate. A more preferred range is from about 5.0% to about 20.0% and most preferably from about 10.0% to about 15.0%.

The - based on the weight of the developer. In one preferred embodiment this component is a mixture of glycerin and \*\*ethylene \*\*glycol\*\* in a relative ratio of about 1:15 to about 200:1.

The developer contains 0.05% to about 5.0% based on the weight of the developer of benzoic or citric acid. A more preferred range is from about 0.1% to about 1.0% and most preferably from about 0.4% to about 0.6%.

The developer contains from about 0.5% to about 10.0% based on the weight of the developer of benzyl alcohol or phenoxyethanol. A more preferred range is from about 1.0% to about 5.0% and most preferably from about 2.0% to about 4.0%. In the preferred embodiment, the developer of the present invention preferably contains optional minor amounts of a conventional antifoam component which aids in processing of the printing plate. This component, when it is used is preferably present in an amount of from about 0.005% to about 0.075% by weight of the developer is observed in these developed plates.

As a comparison, Newplates S plates developed with Graphex Lacquer (available from Graphex litho System of Milwaukee, WI 53214) using the same processor parameters is used as a control. The lithographic properties are given below:

Lithographic Property	This Developer	Comparative Lacquer
Solid/ghost on a 21 step Stouffer	5/9	7/9
Resolution:		
UGRA Circle (microns)	8-12	12-15
UGRA 150 lines per inch	2-98%	1-97%
Impressions to Roll-up	15 +/- .5	30 +/- .5
Length of Run (abrasive conditions)	220M	75M
Redeposit	None	Yes
Odor	Strong	Weak

The experim

----- 4822514  
 classes:1 510/400 1 510/406 1 510/421 1 510/422 1 510/423 1 510/433 1 510/481  
 score: 509

keywords: alkaline;amide;glycol;amide;alkaline;aqueous;acid;amine;glycol;ether;ethylene;

61790-44-1 about 0.8%  
 tall oil  
 Tripropylene \*\*glycol\*\*  
 112-35-6 Dowanol about 3.5%  
 monomethyl \*\*ether\*\* TPM  
 Triethanolamine  
 102-71-6 about 1.5%  
 Coconut diethanolamide  
 68603-42-9  
 Clindrol about  
 100 C-G 0.75%  
 Polyalkoxylated  
 52624-57-4  
 Pluracol V-10  
 about 1.0%  
 trimethylolpropane  
 Disodium EDTA 64-02-8 about  
 0.08%  
 Fragrance about 0.36% P-250  
 about 0.4%  
 about 0.04% Geraniol 500  
 Water Q.S.

While the cleaning and selective gloss or shine imparting or restoring mechanism of the novel composition of the invention is not completely

----- 4248724  
 classes:1 508/208 1 252/387 1 252/396

score: 504

keywords: glycol;glycol ether;ethylene glycol;ethylene glycol;propylene;glycol ether;acid;glycol;ether;ethylene;

- 11,396,52 A,52 R

## References Cited [Referenced By]

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Primary Examiner:Howard; Jacqueline V.

Attorney, Agent or Firm:Krass &amp; Young

## Claims

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A penetrating and lubricating composition consisting essentially of:

- (a) from 0.7% to 1.6% by volume of a dimethyl siloxane polymer; and  
 (b) a \*\*glycol \*\*ether\*\* selected from the group consisting of \*\*ether\*\*s of \*\*ethylene\*\*  
 \*\*glycol\*\*, \*\*ether\*\*s of \*\*propylene\*\* \*\*glycol\*\*, \*\*ether\*\*s of diethylene \*\*glycol\*\*, and  
 \*\*ether\*\*s of dipropylene \*\*glycol\*\*.

2. The composition of claim 1 wherein said \*\*glycol \*\*ether\*\* is \*\*ethylene \*\*glycol\*\*  
 butyl \*\*ether\*\*.

3. The composition of claim 1 wherein said dimethyl siloxane polymer has an  
 average viscosity at 25.degree. centigrade of about 100 centistokes.

4. A penetrating and lubricatin

tion is applied to the exterior of the corroded parts  
 that are to be separated. The extremely low surface tension of the \*\*glycol\*\*  
 \*\*ether\*\* solvent--on the order of from 25 to 30 dynes per centimeter at  
 25.degree. centigrade--allows the formula to seek out and seep into the  
 most minute cracks between the surfaces to be freed and dissolve any rust  
 or corrosion present. The dimethyl siloxane polymer is carried into the  
 cracks with the \*\*glycol \*\*ether\*\* in which it is dissolved and remains on the  
 surfaces as a lubricating and corrosion preventing coating after the  
 \*\*glycol \*\*ether\*\* has evaporated.

For the purposes of this invention, \*\*glycol \*\*ether\*\* will be defined as the  
 \*\*ether\*\*s of \*\*ethylene\*\*, \*\*propylene\*\*, diethylene, or dipropylene \*\*glycol\*\*. These  
 are produced by the reaction of alkylene oxides with alcohols or phenols,  
 the \*\*glycol \*\*ether\*\*s used in the following examples were obtained from the  
 Dow Chemical Company which markets them under the trade name Do

- fluid ounce of Dow Corning 200 fluid of 100 centistokes viscosity (at  
 25.degree. centigrade) was mixed with one gallon of Dowanol EB (ethylene  
 \*\*glycol\*\* butyl \*\*ether\*\*). This formulation was found to have superior  
 penetrating lubricating properties when applied to corroded metallic  
 parts.

## EXAMPLE 2

A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes  
 viscosity and one gallon of Dowanol DE (diethylene \*\*glycol\*\* ethyl \*\*ether\*\*) was  
 made and tested with good results.

## EXAMPLE 3

A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes  
 viscosity and one gallon of Dowanol PM (propylene \*\*glycol\*\* methyl \*\*ether\*\*) was  
 made and tested with good results.

## EXAMPLE 4

A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes  
 viscosity and one gallon of Dowanol DPM (dipropylene \*\*glycol\*\* methyl \*\*ether\*\*)  
 was made and tested with good results.

The above examples are listed in order of declining overall efficiency as  
 pene

----- 5571447

classes:1 510/206 1 252/793 1 430/331 1 510/188

score: 503

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;glycol;propylene glycol;ethylene glycol;hydroxi  
 de;ethylene glycol;propylene glycol;propylene;alkaline;ammonium fluoride;fluoride;buffered;fluoroboric;aq  
 ueous;acid;ammonium;glycol;ethylene;

- /or dimethylsulfoxide.

Advantageously, the compositions of the invention are free of \*\*ethylene\*\*

**\*\*glycol\*\*** and hydrofluoric acid.

The compositions of the invention advantageously contain a corrosion inhibitor.

It is a general object of the invention to provide a stripping composition having a controlled etch and sidewall polymer removal capability.

It is another object of the invention to provide a stripping composition which is free of **\*\*ethylene glycol\*\*** and hydrofluoric acid.

It is yet another object of the invention to provide a stripping composition which is water soluble having high resistance to metal corrosion.

It is still another objection of the invention to provide a method for stripping which provides for a controlled oxide etch.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates that the overall selective etching of the compositions of the invention with respect to **\*\*tetraethyl\*\*orthosilicate** (TEOS) vs. silicon is superior over a wide range

- oxide surfaces. The composition are also effective in removing many forms of resist ash and etch residues without the presence of hydrogen **\*\*fluoride\*\***.

The stripping and cleaning compositions of the invention contain about 60 to 85% by weight of composition of a polyhydric alcohol which excludes **\*\*ethylene glycol\*\***, for example **\*\*propylene glycol\*\***, glycerol, polyethylene **\*\*glycol\*\***, etc.

Fluoboric acid in an amount of about 0.5 to 10% by weight of composition is an essential component of the composition. Fluoboric acid maintains a stable **\*\*fluoride\*\*** ion source with **\*\*ammonium fluoride\*\*** or other **\*\*fluoride\*\*** salts and provides the needed low pH of the compositions.

Polar solvents which can be used in the invention include water, dialkylsulfoxide (dimethylsulfoxide), N-substituted **\*\*pyrrolidone\*\***, aliphatic **\*\*amide\*\***s, aliphatic esters, lactates, cyclic **\*\*lactone\*\***s, and the like.

The use of about 5 to 40% by weight of a polar solvent, especially water and/or dimethyl

----- 4622303

classes:1 435/243 1 435/3 1 435/246 1 435/2557 1 435/812 1 514/937 1 514/943 1 514/945 1 516/134  
score: 500

keywords: glycol;glycol ether;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene glycol ether;aqueous;acid;glycol;ether;ethylene;

- n accordance with claim 8 wherein the polyglycol **\*\*ether\*\***s contain an average of from 1 to 3 **\*\*ethylene glycol ether\*\*** groups.

10. A fermentation broth in accordance with claim 7 wherein from about 0.005 to about 1 g/l of said polyglycol **\*\*ether\*\***s are present in the fermentation broth.

11. A fermentation broth in accordance with claim 10 wherein from about 0.01 to about 0.2 g/l of said polyglycol **\*\*ether\*\***s are present in the fermentation broth.

12. A fermentation broth in accordance with claim 7 wherein the alcohol component of the polyglycol **\*\*ether\*\***s is a mixture of oleyl alcohol and cetyl alcohol components in a ratio by weight of from about 3:1 to about 1:1, and wherein the polyglycol **\*\*ether\*\***s contain an average of 2 **\*\*ethylene glycol ether\*\*** groups.

#### Description

#### BACKGROUND OF THE INVENTION

##### 1. Field of the Invention

The present invention relates to compounds for the suppression of foaming in yeast fermentation without the inhibition of yeast growth or t

- myristyl and stearyl alcohol can also be present in admixture with oleyl alcohol and cetyl alcohol, their quantity should not exceed 10% by weight in either case. Polyglycol **\*\*ether\*\***s in which from about 30 to about 80% of the alcohol component consists of oleyl alcohol are particularly suitable. More highly unsaturated alcohols are unsuitable and should preferably be absent.

The number of polyglycol **\*\*ether\*\*** groups amounts on average to from 1 to 4 and preferably to from 1 to 3. As normal with commercial alkoxylation products, this figure represents a statistical average, i.e. the alkoxylation products may also contain small amounts of alcohols alkoxyated to a higher or lower degree, including unsubstituted alcohols, corresponding to a statistical distribution. The **\*\*glycol ether\*\*** groups can consist of **\*\*ethylene glycol ether\*\*** groups (EO) or **\*\*propylene glycol ether\*\*** groups (PO) or both. They preferably consist of EO-groups; their number



----- 5419779  
 classes:1 134/38 1 134/2 1 134/40 1 252/364 1 252/387 1 252/392 1 252/396 1 510/176 1 510/202 1 510/206 1  
 510/212 1 510/499  
 score: 496

keywords: hydroxide;aqueous;acid;amine;

- e like.  
 Specific examples of organic polymeric materials include positive resists containing phenolformaldehyde resins or poly (p-vinylphenol), polymethylmethacrylate-containing resists, and the like. Examples of plasma processing residues (sidewall polymer) include among others; metal-organic complexes and/or inorganic salts, oxides, \*\*hydroxide\*\*s or complexes which form films or residues either alone or in combination with the organic polymer resins of a photoresist. The organic materials and/or SWP can be removed from conventional substrates known to those skilled in the art, such as silicon, silicon dioxide, aluminum, aluminum alloys, copper, copper alloys, etc. The hydroxylamine utilized in the invention is commercially available as a 50% \*\*aqueous\*\* solution from Nissin Chemical Industry Company, Tokyo, Japan.

The effectiveness and unexpected nature of the stripping compositions of the invention is illustrated, but not lim

----- 6465403  
 classes:1 510/175 1 510/176 1 510/178 1 134/3 1 438/745  
 score: 486

keywords: alkaline;ph;sulfoxide;glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;tetraethyl;tetrame  
 thyl ammonium;tetramethyl;alkaline;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;

- ent  
 \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.1 weight percent trans-(1,2-cyclohexylenedinitrilo)tetraacetic acid (CyDTA), 0.07 weight percent of the non-ionic surfactant Surfynol-465, 0.14 weight percent (calculated as % SiO.sub.2) \*\*tetramethyl\*\*ammonium silicate (TMAS) and 3 weight percent glycerol added with the remainder of this solution being water and has a pH of about 12.1. \*\*aqueous\*\* solution "M1" was prepared with 1.2 weight percent \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.45 weight percent trans-(1,2-cyclohexylenedinitrilo)-tetraacetic acid (CyDTA), 0.14 weight percent (calculated as % SiO.sub.2) \*\*tetramethyl\*\*ammonium silicate (TMAS), 18.5 weight percent hydroxylamine and 0.07 weight percent of the non-ionic surfactant Surfynol-465 (remainder of this solution being water) and has a pH of about 12.1. \*\*aqueous\*\* solution "P1" was prepared with 2.2 weight percent \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.11 weight percent tra

----- 5381807  
 classes:1 134/2 1 134/38 1 134/40 1 134/42 1 430/258  
 score: 480

keywords: amide;glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;amide;  
 acid;glycol;ethylene;

- atoms.  
 Preferred alkanolamines suitable for use in the present invention can be represented by the chemical formula R.sub.1 R.sub.2 --N--CH.sub.2 CH.sub.2 --O--R.sub.3 wherein R.sub.1 and R.sub.2 can be H, CH.sub.3, CH.sub.3 CH.sub.2 or CH.sub.2 CH.sub.2 OH and R.sub.3 is CH.sub.2 CH.sub.2 OH. Examples of suitable alkanolamines include monoethanolamine, diethanolamine, triethanolamine, tertiarybutyldiethanolamine, isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine, 2-amino-(2-ethoxyethanol), and 2-amino(2-ethoxy)propanol. Polar solvents suitable for use in the present invention include \*\*ethylene\*\* \*\*glycol\*\*, ethylene \*\*glycol\*\* alkyl \*\*ether\*\*, diethylene \*\*glycol\*\* alkyl \*\*ether\*\*, triethylene \*\*glycol\*\* alkyl \*\*ether\*\*, \*\*propylene\*\* \*\*glycol\*\*, \*\*propylene\*\* \*\*glycol\*\* alkyl \*\*ether\*\*, dipropylene \*\*glycol\*\* alkyl \*\*ether\*\*, tripropylene \*\*glycol\*\* alkyl \*\*ether\*\*,  
 Page 17

N-substituted \*\*pyrrolidone\*\*, \*\*ethylene\*\*diamine, and \*\*ethylene\*\*triamine.  
Additional polar solvents as known in the a

----- 5498293  
classes:1 134/3 1 134/2  
score: 480

keywords: alkaline;ph;glycol;glycol ether;propylene glycol;ammonium hydroxide;ammonium hydroxide;hydroxid  
e;propylene glycol;propylene;glycol ether;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;fluoride;a  
queous;acid;ammonium;amine;glycol;ether;

- on of a \*\*propylene glycol \*\*ether\*\* organic solvent,  
particularly a \*\*propylene \*\*glycol\*\* monoalkyl \*\*ether\*\* such as n-butoxypropanol,  
is also beneficial to enhance detergency and increase the metal holding  
capacity of the cleaner composition. A preferred formulation will contain  
both a \*\*propylene \*\*glycol\*\* monoalkyl \*\*ether\*\* organic solvent and a metal  
chelating agent, preferably \*\*ethylene\*\*diaminetetraacetic acid and  
n-butoxypropanol.

In the cleaner compositions used in the process of this invention, the  
\*\*alkaline\*\* component will generally be present in an amount of up to about  
25% by weight of the composition, generally in an amount of from about  
0.05 to about 10% by weight, and preferably in an amount of from about 0.1  
to about 5% by weight. The amphoteric surfactant will generally be present  
in an amount of up to about 10% by weight, generally in an amount of from  
about 0.001 to about 10% by weight, and preferably in an amount of from  
about

----- 4836950  
classes:1 510/170 1 510/172 1 510/407 1 510/412 1 510/413 1 510/505 1 510/506  
score: 477

keywords: ph;glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;glycol;et  
her;ethylene;

-		no	no		
	methyl **ether**				
	Ethanol	15.80	8.80	19.40	
				no	no
	Ethyl acetate	15.80	5.30	7.20	yes --
	**ethylene** dichloride				
		19.00	7.40	4.10	yes --
	**ethylene **glycol** mono-				
		15.90	4.70	10.60	
				yes	--
	ethyl **ether** acetate				
	Hexane	14.90	0.00	0.00	no no
	Isophorone	16.90	8.20	7.40	no.sup.b
					yes
	Methanol	15.10	12.30	22.30	
				no	no
	Methylene chloride				
		18.20	6.30	6.10	yes --
	N--methyl-2-pyrro-				
		18.00	12.30	7.20	yes --
	lidone				
	Nitrobenzene	20.00	8.60	4.10	yes --
	2-nitropropane				
		16.20	12.10	4.10	yes --
	**propylene** carbonate				
		20.00	18.00	4.10	no yes
	**propylene **glycol** mono-				
		15.60	6.3		

----- 6326348  
classes:1 510/428 1 510/119 1 510/122 1 510/125 1 510/235 1 510/311 1 510/357 1 510/424 1 510/426 1 510/4  
27 1 510/505 1 510/506  
score: 449

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd

roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- queous liquid phase is one selected from the alkali metal salts of alkylbenzene sulfonic acids in which the alkyl group contains from about 10 to 16 carbon atoms, in straight chain or branched chain configuration. (See U.S. Pat. Nos. 2,220,099 and 2,477,383, incorporated herein by reference.) Especially preferred are the sodium and potassium linear straight chain alkylbenzene sulfonates (LAS) in which the average number of carbon atoms in the alkyl group is from about 11 to 14. Sodium C.sub.11 -C.sub.14 LAS is especially preferred.
- The alkylbenzene sulfonate anionic surfactant will be dissolved in the nonaqueous liquid diluent which makes up the second essential component of the nonaqueous phase. To form the structured liquid phase required for suitable phase stability and acceptable rheology, the alkylbenzene sulfonate anionic surfactant is generally present to the extent of from about 30% to 65% by weight of the liquid phase.
- erein, i.e., the peroxygen bleaching agents, sodium perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.
- A preferred type of nonaqueous, low-polarity solvent for use in the compositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylenediol and 1,4-butylenediol. Hexylene \*\*glycol\*\* is the most preferred.
- Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.s

----- 5880062

classes:1 503/201 1 347/56 1 347/98 1 347/105 1 427/152 1 503/205 1 503/206  
score: 449

keywords: ph;glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;tetraethyl;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- xture of water and other ink components, the concentration of desensitizing agent may be limited to the amount soluble in the ink. Typical concentrations of desensitizing agent in the ink are at least about 0.5 percent by weight, typically from about 1 to about 40 percent by weight, preferably from about 2 to about 20 percent by weight, more preferably from about 2 to about 10 percent by weight, although the amount can be outside these ranges.
- The liquid vehicle of the inks of the present invention may consist of water, or it may comprise a mixture of water and a miscible organic component, such as \*\*ethylene glycol\*\*, \*\*propylene glycol\*\*, diethylene glycol, glycerine, dipropylene glycol, polyethylene glycol, polypropylene glycol, \*\*amide\*\*s, such as urea and substituted ureas, \*\*ether\*\*s, carboxylic acids, esters, alcohols, organosulfides, organosulfoxides, \*\*sulfone\*\*s, alcohol derivatives, carbitol, butyl carbitol, cellusolve, \*\*ether\*\*de

----- 4239661

classes:1 438/471 1 134/2 1 134/38 1 134/42 1 430/326 1 430/331 1 438/329 1 438/745 1 438/754 1 510/175 1 510/372 1 510/373 1 510/421 1 510/434 1 510/504  
score: 447

keywords: alkaline;ph;glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;alkaline;aqueous;acid;ammonium;glycol;ether;

- material of a positive-working photoresist in an organic solvent. The photoresist film was heated about 20 minutes at 80.degree. C. to evaporate the organic solvent and to suppress fogging. Later, a light was projected through a prescribed photomask aligned with the photoresist. Those portions of the photoresist on which a light was projected were etched by a 2.44% \*\*aqueous\*\* choline solution while those portions of the photoresist

on which a light was not projected were left, thereby causing the surface of the silicon oxide film to be selectively exposed. An image pattern produced had a distinctly defined outline and a high precision.

The above-mentioned intermediate product was again heated 20 minutes at 120.degree. C. to attain a closer attachment between the silicon oxide film and the remaining portion of the photoresist film. Later, the exposed silicon oxide film was etched out with dilute hydrofluoric acid. Those portion

----- 6015781

classes:1 510/302 1 510/276 1 510/367 1 510/421 1 510/422 1 510/506  
score: 442

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hydroxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- li metal salts of alkylbenzene sulfonic acids in which the alkyl group contains from about 10 to 16 carbon atoms, in straight chain or branched chain configuration. (See U.S. Pat. Nos. 2,220,099 and 2,477,383, incorporated herein by reference). Especially preferred are the sodium and potassium linear straight chain alkylbenzene sulfonates (LAS) in which the average number of carbon atoms in the alkyl group is from about 11 to 14. Sodium C.sub.11 -C.sub.14 LAS is especially preferred.
- The alkylbenzene sulfonate anionic surfactant will be dissolved in the nonaqueous liquid diluent which makes up the second essential component of the nonaqueous phase. To form the structured liquid phase required for suitable phase stability and acceptable rheology, the alkylbenzene sulfonate anionic surfactant is generally present to the extent of from about 30% to 65% by weight of the liquid phase. More preferably, the alkylbenzene sulfonate
- um perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.
- A preferred type of nonaqueous, low-polarity solvent for use in the compositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred.
- Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\* mono C.sub.2 -C.sub.6 a

----- 6008181

classes:1 510/426 1 510/424 1 510/428 1 510/506 1 560/76 1 568/458 1 568/882  
score: 440

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hydroxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- erial be capable of actually dissolving all of the detergent composition components added thereto.
- The nonaqueous organic materials which are employed as solvents herein are those which are liquids of low polarity. For purposes of this invention, "low-polarity" liquids are those which have little, if any, tendency to dissolve one of the preferred types of particulate material used in the compositions herein, i.e., the peroxygen bleaching agents, sodium perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.
- A preferred type of nonaqueous, low-polarity solvent for use in the

C  
 - compositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred.  
 Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\* mono C.sub.2 -C.sub.6 alkyl \*\*ether\*\*s. The specific examples of such compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monoethyl \*\*ether\*\*, and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*. Diethylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve.

Another preferred type of nonaqueous, low-polarity organic solvent usefu

----- 6060443  
 classes:1 510/426 1 510/424 1 510/428 1 560/76 1 568/458 1 568/882  
 score: 436

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd  
 roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;a  
 cid;ammonium;amine;glycol;ether;ethylene;

- e specific examples of such compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monoethyl \*\*ether\*\*, and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*. Diethylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve.

Another preferred type of nonaqueous, low-polarity organic solvent useful herein comprises the lower molecular weight polyethylene \*\*glycol\*\*s (PEGs). Such materials are those having molecular weights of at least about 150. PEGs of molecular weight ranging from about 200 to 600 are most preferred.  
 Yet another preferred type of non-polar, nonaqueous solvent comprises lower molecular weight methyl esters. Such materials are those of the general formula: R.sup.1 --C(O)--OCH.sub.3 wherein R.sup.1 ranges from 1 to about 18. Examples of sui

----- 6153577  
 classes:1 510/356 1 510/505 1 510/506  
 score: 436

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd  
 roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;a  
 cid;ammonium;amine;glycol;ether;ethylene;

- itions are higher molecular weight nonionics, such as Neodol 45-11, which are similar \*\*ethylene\*\* oxide condensation products of higher fatty alcohols, with the higher fatty alcohol being of 14-15 carbon atoms and the number of \*\*ethylene\*\* oxide groups per mole being about 11. Such products have also been commercially marketed by Shell Chemical Company.

The alcohol alkoxylate component which is essentially utilized as part of the liquid diluent in the nonaqueous compositions herein will generally be present to the extent of from about 1% to 60% of the liquid phase composition. More preferably, the alcohol alkoxylate component will comprise about 5% to 40% of the liquid phase. Most preferably, the essentially utilized alcohol alkoxylate component will comprise from about 5% to 30% of the detergent composition liquid phase. Utilization of alcohol alkoxylate in these concentrations in the liquid phase corresponds to an alcohol alk

- branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred.

Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\* mono C.sub.2 -C.sub.6 alkyl \*\*ether\*\*s. The specific examples of such

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compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monoethyl \*\*ether\*\*, and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*. Diethylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve.

Another preferred type of nonaqueous, low-polarity organic solvent useful herein comprises the lower molecular weight polyethylen

----- 6448213  
classes:1 510/357 1 510/424 1 510/426 1 510/428 1 568/458 1 560/76  
score: 433

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;pyrrolidone;hydroxide;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

-	W				
	LAS	14	14	14	
	C45 AS	2.4	2.4	2.4	
	C45E1S	0.9	0.9	0.9	
	C23E6.5	1.5	1.5	1.5	
	C16 branched sulfate, sodium salt	8.0	--	--	
	C17 branched sulfate, sodium salt	--	8.0	4.0	
	C18 branched sulfate, sodium salt	--	--	4.0	
	QAS	1.5	1.5	1.5	
	Zeolite A	26	26	26	
	Carbonate	19.3	19.3	19.3	
	Sulfate	5	5	5	
	Perborate	1	1	1	
	Polyacrylic Acid (MW = 4500)	2	2	2	
	Polyethylene **glycol** (MW = 4600)	0.9	0.9	0.9	0.9
	Silicate	0.6	0.6	0.6	
	Water	--	--	--	Balance

-		AA	BB	CC	
	MBAS (14.5-15.5 ave. total carbon)	7.5	11	14	
	C11.3 LAS	14	11	7.5	
	QAS	1	1	1	
	LMFAA	2.5-3.5	2.5-3.5	2.5-3.5	
	C23E9	0.6-2	0.6-2	0.6-2	
	APA	0-0.5	0-0.5	0-0.5	
	Citric Acid	3.0	3.0	3.0	
	Fatty Acid (TPK or C12/14)	2.0	2.0	2.0	
	Ethanol	3.4	3.4	3.4	
	Propanediol	6.4	6.4	6.4	
	Monoethanol **amine**	1.0	1.0	1.0	
	NaOH	3.0	3.0	3.0	
	Na toluene sulfonate	2.3	2.3	2.3	
	Na formate	0.1	0.1	0.1	
	Borax	2-2.5	2-2.5	2-2.5	
	P				

-	EE	FF	
	MBAS (14.5-15.5 ave. total carbon)	13	10 7
	C11.3 LAS	7	10 13
	Any combination of:	1	1 1
	C25 AExS*Na (x = 1.8-2.5)		
	C25 AS (linear to high 2-alkyl)		
	C14-17 NaPS		
	C12-16 SAS		
	C18 1,4 disulfate		
	C12-16 MES		
	QAS	1	1 1
	LMFAA	3.5-5.5	3.5-5.5 3.5-5.5
	C23E9	4-6	4-6 4-6
	APA	0-1.5	0-1.5 0-1.5
	Citric Acid	1	1 1
	Fatty Acid (TPK or C12/14)	7.5	7.5 7.5
	Fatty Acid (Rapeseed)	3.1	3.1 3.1
	Ethanol	1.8	1.8 1.8
	Propanediol	9.4	9.4 9.4
	Monoethanol **amine**	6.5	6.5 6.5
	NaOH		

----- 6228829  
 classes:1 510/357 1 510/424 1 510/426 1 510/428 1 560/76 1 568/458 1 568/882  
 score: 433

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;pyrrolidone;hydroxide;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

----- 6306812  
 classes:1 510/310 1 8/111 1 8/137 1 134/252 1 134/42 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302 1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376  
 score: 432

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- olute ethanol (100 mL) and this solution is refluxed 1 hour. During that time, a tan solid formed which is separated from the mother liquor by vacuum filtration using Whatman #1 filter paper. The solid is dried under vacuum, overnight. Yield: 1.79 g, II, (15%). Fab Mass Spec. TG/G, MeOH) M.sup.+ 266 mu, 60%, MI.sup.+ 393 mu, 25%.  
 Synthesis of 5,8 Dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane  
 To a stirred solution of II, (1.78 g, 3.40 mmol) in ethanol (100 mL, 95%) is added sodium borohydride (3.78 g, 0.100 mmol). The reaction is stirred under nitrogen at RT for 4 days. 10% Hydrochloric acid is slowly added until the pH is 1-2 to decompose the unreacted NaBH.sub.4. Ethanol (70 mL) is then added. The solvent is removed by roto-evaporation under reduced pressure. The product is then dissolved in \*\*aqueous\*\* KOH (125 mL, 20%), resulting in a pH 14 solution. The product is then extracted with benzene (5.times.60 mL) and the combined

----- 6242406  
 classes:1 510/357 1 510/424 1 510/426 1 510/427 1 510/428 1 510/473  
 score: 432

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hydroxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

-	Zeolite A	260	260	260
	Methyl Cellulose	10	20	5
	Sodium Carbonate	193	193	193
	Sodium Sulfate	52	52	52
	Sodium Perborate	10	10	10
	Polyacrylic Acid (MW = 4500)	22	22	22
	Polyethylene **glycol** (MW = 4600)	9	9	9
	Sodium Silicate	6	6	6

#### EXAMPLE 15

The following laundry detergent compositions A to I are prepared in accord with the invention:

	A	B	C	D	E	F	G	H	I
LAS	10	10	10	20	20	20	0	0	0
C45 AS	10	10	10	0	0	0	20	20	20
MBAE	1	2.5	5	1	2.5	5	1	2.5	5
Zeolite A	28	28	28	28	28	28	28	28	28
PAA	2								

-			W	X	Y	Z
	MBAEx (x = 5-10; 14.6-15.5 ave. total carbon in alkyl group)	0.5-5	4-6	10-15	20-25	
	Any combination of: C25 AExS*Na (x = 1.8- 2.5)	21.5	19	5-15	1-6	
	C25 AS (linear to high 2-alkyl)					

C14-17 NaPS  
 C12-16 SAS  
 C18 1,4 disulfate  
 C12-16 MES  
 C11.3 LAS                    5                    1                    10                    15  
 LMFAA                    2.5-5.5                    2.5-5.5                    0-3                    0-3  
 Any combination of:    0-1.5                    0-1.5                    0-2                    0-3  
 APA  
 QAS  
 C12-14 trimethyl  
 \*\*ammonium\*\* halide  
 DSDMAC                    --                    --                    --                    4  
 Methyl Cellulose                    3                    3                    1                    5  
 Citric Acid                    3                    1                    1                    1  
 Fatty Acid (TPK, C12/14                    2                    10.6                    0-5                    0-5  
 or Rapeseed)  
 Ethanol                    3.4                    1.8                    4                    5.5  
 Propaneidol                    6.4                    9.4                    6

----- 6399557

classes:1 510/310 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302 1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376  
 score: 430

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- he oil is  
 taken up in absolute ethanol (100 mL) and this solution is refluxed 1 hour. During that time, a tan solid formed which is separated from the mother liquor by vacuum filtration using Whatman #1 filter paper. The solid is dried under vacuum, overnight. Yield: 1.79 g, II, (15%). Fab Mass Spec. TG/G, MeOH) M.sup.+ 266 mu, 60%, M.sup.+ 393 mu, 25%.

Synthesis of 5,8

Dimethyl-1,5,8,12-tetraazabicyclo.lambda.10.3.2]heptadecane  
 To a stirred solution of II, (1.78 g, 3.40 mmol) in ethanol (100 mL, 95%) is added sodium borohydride (3.78 g, 0.100 mmol). The reaction is stirred under nitrogen at RT for 4 days. 10% Hydrochloric acid is slowly added until the pH is 1-2 to decompose the unreacted NaBH.sub.4. Ethanol (70 mL) is then added. The solvent is removed by roto-evaporation under reduced pressure. The product is then dissolved in \*\*aqueous\*\* KOH (125 mL, 20%), resulting in a pH 14 solution. The product is then extracted with benzen

----- 4102823

classes:1 510/351 1 510/348 1 510/352 1 510/355 1 510/452 1 510/453 1 510/488  
 score: 428

keywords: alkaline;ph;alkaline;aqueous;acid;ethylene;

- erformance quality, physical  
 characteristics and processability are prepared if, in the above-described Example IV composition, the sodium tripolyphosphate is replaced with an equivalent amount of potassium tripolyphosphate, potassium hexametaphosphate, tetrapotassium pyrophosphate or sodium pyrophosphate.

A composition of substantially similar performance quality, physical characteristics and processability is prepared if, in the above-described Example IV composition the hydrogenated fish oil fatty acid is replaced with an equivalent amount of tallow fatty acid or mixtures of tallow fatty acid and hydrogenated fish oil fatty acid containing about 18 carbon atoms.

#### WASH-WEAR TEST

Detergency performance of the instant invention is compared with that of a commercially-available built granular laundry detergent in a wash and wear test. The test employed is conducted in the following manner: Light-colored dress shi

----- 6306817

classes:1 510/535 1 510/357 1 510/424 1 510/426 1 510/428



score: 427

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;hydroxide;ethylene glycol;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- NOVO Industries A/S

MEA Monoethanolamine

PG Propanediol

EtOH Ethanol

NaOH Solution of sodium \*\*hydroxide\*\*

NaTS Sodium toluene sulfonate

Citric acid Anhydrous citric acid

CxyFA C.sub.1x --C.sub.1y fatty acid

CxyEz A C.sub.1x-1y branched primary alcohol condensed with an average of 2 moles of \*\*ethylene\*\* oxide

Carbonate Anhydrous sodium carbonate with a particle size between 200 .mu.m and 900 .mu.m

Citrate Tri-sodium citrate dihydrate of activity 86.4% with a particle size distribution between 425 .mu.m and 850 .mu.m

TFAA C16-18 alkyl N-methyl glucamide

LMFAA C12-14 alkyl N-methyl glucamide

APA C8-C10 amido propyl dimethyl \*\*amine\*\*

Fatty Acid C12-C14 fatty acid (C12/14)

Fatty Acid Topped palm kernel fatty acid (TPK)

Fatty Acid Rapeseed fatty acid (RPS)

Borax Na tetraborate decahydr

size in the range from 0.1 to 10 micrometers

NaSKS-6 Crystalline layered silicate of formula .delta.-Na.sub.2 Si.sub.2 O.sub.5

Bicarbonate Anhydrous sodium bicarbonate with a particle size distribution between 400 .mu.m and 1200 .mu.m

Silicate Amorphous Sodium Silicate (SiO.sub.2 :Na.sub.2 O; 2.0 ratio)

Sulfate Anhydrous sodium sulfate

PAE ethoxylated \*\*tetraethyl\*\*ene pentamine

PIE ethoxylated polyethylene imine

PAEC methyl quaternized ethoxylated dihexylene triamine

MA/AA Copolymer of 1:4 maleic/acrylic acid, average molecular weight about 70,000.

CMC Sodium carboxymethyl cellulose

Protease Proteolytic enzyme of activity 4KNPU/g sold by NOVO Industries A/S under the tradename Savinase

Cellulase Cellulytic enzyme of activity 1000 CEVU/g sold by NOVO Industries A/S under the tradename Carezyme

Amylase

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classes:1 510/311 1 510/376 1 510/500

score: 425

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- 3 3

Perfume 0.5 3 1 0.5 2 1.00

Soil Release 0.50 0.75 0.75 0.75 0.50 0.75

Polymer.sup.5

Product of 2.5 10 5 0.5 1 20

Example.sup.6 ppm ppm ppm ppm ppm ppm

Water to 100 to 100 to 100 to 100 to 100 to 100

.sup.1 Di-(soft-tallowyloxyethyl) dimethyl \*\*ammonium\*\* chloride or Distearyl dimethyl ammonium chloride

.sup.2 Diethylenetriamine Pentaacetic acid(3) DC-2310, sold by Dow-Corning

.sup.3 DC-2310, sold by Dow-Corning

.sup.4 Kathon CG, sold by Rohm & Has

.sup.5 Copolymer of \*\*propylene\*\* terephthalate and \*\*ethylene\*\*oxide

.sup.6 Mn(Bcyclam)Cl.sub.2 as in Synthesis Example 1

## EXAMPLE 27

##STR56##

Synthesis of 1,5,9,13-Tetraazatetracyclo[1.2.2.2.sup.5.9]heptadecane  
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in  
acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,  
40% a

----- 6387862  
classes:1 510/311 1 510/376 1 510/500  
score: 425

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- lease 0.50 0.75 0.75 0.75 0.50 0.75  
Polymer.sup.5  
Product of 2.5 10 5 0.5 1 20  
Example.sup.6 ppm ppm ppm ppm ppm ppm  
Water to 100 to 100 to 100 to 100 to 100 to 100  
.sup.1 Di-(soft-tallowyloxyethyl) dimethyl \*\*ammonium\*\* chloride or  
Distearyldimethylammonium chloride  
.sup.2 Diethylenetriamine Pentaacetic acid(3) DC-2310, sold by Dow-Corning  
.sup.3 DC-2310, sold by Dow-Corning  
.sup.4 Kathon CG, sold by Rohm & Has  
.sup.5 Copolymer of \*\*propylene\*\* terephthalate and \*\*ethylene\*\*oxide  
.sup.6 Mn(Bcyclam)Cl.sub.2 as in Synthesis Example 1

## EXAMPLE 27

Dithiocyanato Manganese (II)

5,8 Dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane Synthesis

##STR54##

Synthesis of 1,5,9,13-Tetraazatetracyclo[11.2.2.2.sup.5,9]heptadecane  
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in  
acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,  
40% aqu

----- 6280527  
classes:1 134/2 1 134/38  
score: 420

keywords: alkaline;ph;glycol;glycol ether;ammonium hydroxide;ammonium hydroxide;hydroxide;glycol ether;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;aqueous;acid;ammonium;glycol;ether;

- clude polar,  
non-polar, hydrophilic, and hydrophobic types of pastes from stencil  
masks, typically Mo metal, electroform masks, and emulsion masks.  
Tetramethyl \*\*ammonium \*\*hydroxide\*\* (TMAH) is the preferred organic base for  
mask cleaning applications according to this invention but other related  
commonly known quaternary \*\*ammonium \*\*hydroxide\*\*s that may also be used are:  
\*\*tetraethyl ammonium \*\*hydroxide\*\*, tetrabutyl \*\*ammonium \*\*hydroxide\*\*,  
trimethyl-2-hydroxyethyl \*\*ammonium \*\*hydroxide\*\* (Choline), triethyl-2-hydroxy  
ethyl \*\*ammonium \*\*hydroxide\*\*, ethyltrimethyl \*\*ammonium \*\*hydroxide\*\*, and the like.  
The \*\*aqueous alkaline\*\* cleaning solution comprising quaternary \*\*ammonium\*\*  
\*\*hydroxide\*\*s may optionally contain one or more corrosion inhibitor.  
According to a preferred embodiment of this invention, a pressurized spray  
wash at between about 50 to about 200 psi with a heated \*\*aqueous\*\* solution  
of \*\*tetramethyl ammonium \*\*hydroxide\*\* (TMAH) containing between about 0.3 and  
about 2.0

----- 5565136  
classes:1 510/203 1 510/206 1 510/212 1 510/418  
score: 414

keywords: glycol;glycol ether;propylene glycol;nmp;ammonium hydroxide;ammonium hydroxide;hydroxide;propylene glycol;propylene;glycol ether;nmp;acid;ammonium;amine;glycol;ether;

- such as \*\*ammonium \*\*hydroxide\*\* and  
monoethanol \*\*amine\*\*, and acidic type such as formic acid, acetic acid and

oxalic acid are not necessary and preferably not included in the compositions of the present invention.

The water-based coating remover of the present invention is useful for removing various cured and uncured paints, varnishes, lacquers and other coatings or finishes, particularly coatings used on wood such as acrylic latex type enamel, polyurethane based clear protective coatings, oil-based alkyl resin, epoxy paint and varnish/lacquer.

The water-based coating remover of the present invention can be used for removing paint, lacquers, varnishes and other coatings or finishes from a variety of substrates, including paint brushes, without significant damage, such as etching, pitting or corrosion to such substrate. Examples of substrates on which the coating remover of the present invention can be used includes: wood; glass; ac

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classes:1 585/7 1 562/93 1 562/95 1 585/10 1 585/11 1 585/18 1 585/19 1 585/24 1 585/323 1 585/329 1 585/455 1 585/467 1 585/533

score: 413

keywords: propylene;aqueous;acid;ammonium;amine;ethylene;

- XVII

In accordance with this Example, benzene was alkylated with an olefin mixture over the dealuminized mordenite prepared in Example XVI. The olefin mixture was \*\*propylene\*\* tetramer (1.3 methyl branches per chain) prepared by oligomerizing \*\*propylene\*\* over a ZSM-23 catalyst having surface acid sites inactivated with 2,4,6-collidine and separating out the C.sub.12 fraction. This olefin mixture was percolated through activated alumina prior to use. To an oven-dried flask under N.sub.2 was added 10 ml perked C.sub.12 olefin (0.05 mol), 50 ml benzene (0.56 mol) and 0.6 g dealuminized mordenite. After 6 hr., GC showed about 10% reaction. Another 0.5 g catalyst was added and the mixture was refluxed 64 hrs. GC showed about 90% reaction. Another 0.25 g catalyst was added and the mixture was refluxed an additional 24 hr. The reaction was cooled, filtered, and evaporated to 10.15 g product, which still contained 4% unreacted olefin.

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classes:1 585/533 1 585/520

score: 409

keywords: propylene;aqueous;acid;ammonium;ethylene;

- hours. The resulting catalyst had an alpha value of 29 and a surface acidity less than 0.1.

Example 4

Surface Acidity Reduction with Collidine

A third sample of 7.7 g of the ZSM-23 catalyst from Example 1 having a silica/alumina ratio of 110:1 was treated with 0.059 g collidine/g of catalyst by contacting the catalyst with a dilute collidine-pentane solution containing 0.0454 g collidine which is an amount sufficient to poison 25% of acid sites present in the zeolite component of the catalyst. The collidine adsorbed very rapidly and completely to the catalyst. The catalyst was dried slowly and then purged with nitrogen.

Example 5

Oligomerization \*\*propylene\*\* with ZSM-23 at 225.degree. C.

The catalysts of Examples 1, 2, 3 and 4 were evaluated in a semi-batch autoclave for activity, product selectivity, and product structure with \*\*propylene\*\* feed on demand for five hours at 800 psig and 225.degree. C. Under the above conditions, unmodified

- ethyls/C.sub.12 necessary

for preparing desirable chemical intermediates. The oxalic acid treated catalyst did not require treatment with collidine to achieve the desired product quality.

The determination of Branching Index is a useful and sensitive method practiced by those skilled in the arts to which the present invention applies and used to quantitatively assess the degree of linearity of a molecule or molecular mixture. The index is determined as follows: the C6 and C9 oligomers are first removed from the sample and the C12+ fraction is hydrogenated using Pd/charcoal catalyst in acetic acid. The

hydrogenated sample is extracted from the acetic acid into deuteriochloroform and the  $^1\text{H}$  NMR spectrum determined. The branching index is defined as the ratio of the intensity (area) of the resonance due to  $\text{CH}_3$  (0.7-1.0 ppm) divided by the sum of the intensities (areas) of the resonances due to  $\text{CH}_3$  (0.7-1.0 ppm) and  $\text{CH}_2$  (1.1-

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classes:1 510/175 1 134/2 1 134/40 1 510/108 1 510/245 1 510/254 1 510/434 1 510/489 1 510/492 1 510/505  
score: 365

keywords: alkaline;amide;glycol;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol;propylene;amide;alkaline;acid;ammonium;glycol;ethylene;

- onoamines. The alkanol group of the **amine**s preferably has from 1 to 5 carbon atoms. Preferred alkanolamines suitable for use in the present invention can be represented by the chemical formula  $\text{R.sub.1 R.sub.2 --N--CH.sub.2 CH.sub.2 --O--R.sub.3}$  wherein  $\text{R.sub.1}$  and  $\text{R.sub.2}$  can be H,  $\text{CH.sub.3}$ ,  $\text{CH.sub.3}$   $\text{CH.sub.2}$  or  $\text{CH.sub.2 CH.sub.2 OH}$  and  $\text{R.sub.3}$  is  $\text{CH.sub.2 CH.sub.2 OH}$ . Examples of suitable alkanolamines include monoethanolamine, diethanolamine, triethanolamine, tertiarybutyldiethanolamine, isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine, 2-amino-2-ethoxyethanol, and 2-amino-2-ethoxy-propanol.
- Polar solvents suitable for use in the stripping composition of the present invention include **ethylene glycol**, **ethylene glycol alkyl ether**, diethylene glycol alkyl ether, triethylene glycol alkyl ether, **propylene glycol**, **propylene glycol alkyl ether**, dipropylene glycol alkyl ether, tripropylene glycol alkyl ether, N-su
- like salts would not therefore be suitable for use based upon the understood mechanism of ionic contamination in a microcircuit as caused by cleaning and set forth above. As evident from the above formula, the carboxylic acid may be mono-, di- or trisubstituted rather than tetra-substituted.
- A preferred cleaning composition of the present invention includes 30% by weight hydroxylamine, 25% by weight 2-amino-2-ethoxyethanol, 5% by weight 1,2-dihydroxybenzene and 50% by weight of water.
- The stripping compositions of the present composition are effective in removing a wide range of positive photoresists but are particularly useful in removing photoresists commonly consisting of an ortho-naphthoquinone diazide sulfonic acid ester or **amide** sensitizer with novolak-type binders or resins. Examples of commercially available photoresist compositions which the stripping compositions of the present invention effectively remove from a su

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classes:1 560/99 1 508/496 1 524/314 1 560/76 1 560/97 1 560/190 1 560/202 1 560/204 1 568/909  
score: 221

keywords: weak;propylene;acid;ethylene;

- ic anhydride (22.2 g, 0.15 mole), tridecanol (60.11 g, 0.3 mole, containing 1.4 branching methyl groups), and p-toluene sulfonic acid (0.1 g). The reaction is heated under reflux for 20 hours. Three milliliters of water is collected. Removing the solvent under a reduced pressure gave 78.2 g (95%) of ditridecylphthalate, a yellow liquid whose viscosity at 40.degree. C. is 43.5 centistokes.
- In comparison, isotridecyl alcohol, which contains 3-4 branching methyl groups, and phthalic anhydride give a product whose viscosity at 40.degree. C. is 83 centistokes. These data show that the more linear alcohol gives a product with improved low temperature properties. Also, one skilled in the art recognizes that more linear alcohols decrease the volatility of esters such as this phthalate. These properties are useful for plasticizing PVC and other plastics. Ditridecylphthalate is also useful as a high temperature lubricant, and finds
- ant properties for some plasticizers.

Procedure for Ethoxylating Tridecanols. This procedure follows those by Satkowski and Hsu, and a Shell Chemical Company Bulletin, see Ind. Eng. Chem.; Satkowski, W. B., Hsu, C. G., "Polyoxyethylation of Alcohol", 1957,

49, 1975, and describes synthesizing a tridecanol-ethoxylate containing nine \*\*ethylene\*\* oxide residues. A 500 mL autoclave is charged with tridecanol [50 g (approximately 60 mL), 0.25 mole] and potassium \*\*hydroxide\*\* (0.15 g, 0.00268 mole), and heated at 135.degree. C. under vacuum (15 mm Hg or lower) for one hour. This removes water. Nitrogen gas is added until the vessel's pressure is 45 psig. Liquid \*\*ethylene\*\* oxide [99 g (113 mL), 2.25 mole] is fed on pressure demand from a calibrated, nitrogen gas blanketed Jurgenson gauge which is fitted with a check valve. \*\*ethylene\*\* oxide flowed once the reactor's pressure dropped below the pressure of the nitrogen gas blanket. Temperatu

Orleans, May 1981. Also, Shell Chemical Co. Technical Bulletin SC:580.82. The above-references are hereby incorporated by reference. An acetylation reagent is prepared by slowly adding acetic anhydride (120 mL) with stirring to a solution of p-toluene sulfonic acid monohydrate (14.4 g) and ethyl acetate (360 mL). This reagent is stored in a tightly sealed bottle shielded from light. A 100 mL flask, equipped with a magnetic spin bar and drying tube, is purged with nitrogen gas. Four grams of alcohol-ethoxylate is dissolved in 4 mL of toluene and transferred to the flask along with 12 mL of acetylation reagent. This solution is heated at 50.degree. C. for 30 minutes, transferred to a separatory funnel along with 40 mL of toluene, and washed three times: 100 mL of 30 wt % brine; 100 mL of bicarbonate-brine (50 g sodium bicarbonate, 250 g sodium chloride, 1750 g water); 100 mL of 30 wt % brine. The toluene solution is dried